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(54) **Biaxially oriented polyester film for magnetic recording media.**

(57) A biaxially oriented polyester film for magnetic recording media, which comprises :  
(A) 0.05 to 1.0 % by weight of  $\theta$ -aluminum oxide or mixed ( $\alpha + \gamma$ ) aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and  
(B) 0.01 to 1.5 % by weight of inert particles having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the aluminum oxide.

## Detailed Description of the Invention

The present invention relates to a biaxially oriented polyester film for magnetic recording media. More specifically, it relates to a biaxially oriented polyester film for magnetic recording media, which is excellent in a variety of performances required of films for magnetic recording media, particularly abrasion resistance and scratch resistance.

Biaxially oriented polyester films typified by a polyethylene terephthalate film are extensively used for magnetic recording media owing to their excellent physical and chemical properties.

In biaxially oriented polyester films, their lubricity and abrasion resistance are crucial factors which influence not only the quality of accomplishment in the manufacturing and processing step but also their product quality.

When the biaxially oriented polyester film is poor in the above properties, for example, abrasion dust occurs or the film surface is scratched due to strong friction between a coating roll and the film surface during the formation of a magnetic tape by coating a magnetic layer on a biaxially oriented polyester film. Further, when such a magnetic tape is used as a VTR or data cartridge, abrasion dust or scratch also occurs when the tape is taken into a cassette at a high speed, which might cause drop-out of recorded data.

In the recent VTR cassette field, a metal guide of which the surface finish is insufficient or a plastic guide is sometimes used as a guide post fixedly provided in the cassette with the intention of reducing the manufacturing cost. This guide post has a very rough surface. Therefore, in a conventional method for improvement in lubricity and abrasion resistance, e.g., a method in which inorganic particles of silicon oxide, titanium dioxide, calcium carbonate, talc, clay, calcined kaolin, etc., are incorporated (see Japanese Laid-Open Patent Publication No. 57762/1979), or a method in which fine particles containing calcium, lithium or phosphor are precipitated in the polymerization system (see Japanese Patent Publication No. 32914/1977), the resultant magnetic tape provided with no back coating causes abrasion dust or scratch when the tape is taken into a cassette at a high speed, and recorded data is often dropped out.

U.S. Patent 5,006,589 discloses a polyester film for magnetic recording media which contains 0.05 to 5 % by weight of inactive inorganic particles (A) having a Mohs hardness of 6 or above and an average particle size of 0.005 to 1.0  $\mu\text{m}$  and 0.01 to 2 % by weight of inactive particles (B) having an average particle size which is greater than that of the particles (A) and in the range of 0.1 to 3.0  $\mu\text{m}$ , the polyester film having an intrinsic viscosity in the range of 0.52 to 0.65.

As a material for the above inactive inorganic particles (A),  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiC}$ , zeolite, sellaite,  $\text{TiC}$  and titanium black are disclosed. Further, it is described that  $\text{Al}_2\text{O}_3$  is particularly preferred, and there are disclosed  $\alpha$ —,  $\beta$ —,  $\gamma$ —,  $\kappa$ —,  $\sigma$ —,  $\delta$ —,  $\eta$ —,  $\chi$ — and  $\rho$ —type  $\text{Al}_2\text{O}_3$ 's.

The above polyester film shows an effect on the guide posts employed for reducing in the manufacturing cost. However, when a conventional metal guide having a well-finished surface is used, the effect on improvement is insufficient. There is also a method in which inactive particles having a little greater size than the above inactive inorganic particles (A) are used in combination to overcome scratch caused by a metal guide having a well-finished surface. In this method, however, there is observed an adversary influence that abrasion dust newly occurs.

It is therefore an object of the present invention to provide a biaxially oriented polyester film for magnetic recording media.

It is another object of the present invention to provide a biaxially oriented polyester film for magnetic recording media, which is excellent in abrasion resistance and scratch resistance against not only a conventional metal guide having a well-finished surface but also a recent metal guide having a rough surface and a recent plastic guide, and which is also excellent in lubricity.

The other objects and advantages of the present invention will be apparent from the following description.

According to the present invention, the above objects and advantages of the present invention are achieved by a biaxially oriented polyester film for magnetic recording media, which comprises:

(A) 0.05 to 1.0 % by weight of  $\theta$ -aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and

(B) 0.01 to 1.5 % by weight of inert particles having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the  $\theta$ -aluminum oxide.

Fig. 1 is a schematic view of an apparatus for measuring a running friction coefficient of a biaxially oriented polyester film for magnetic recording media.

Fig. 2 is a projection distribution curve.

The polyester which constitutes the biaxially oriented polyester film of the present invention is preferably selected from aromatic polyesters comprising aromatic dicarboxylic acids as a main acid component and aliphatic glycols having 2 to 10 carbon atoms as a main glycol component. As such a polyester, polyesters which are

substantially linear and have film formability in the melt-molding are advantageous.

Examples of the aromatic dicarboxylic acids are terephthalic acid, naphthalenedicarboxylic acid, isophthalic acid, diphenoxyethanedicarboxylic acid, diphenyldicarboxylic acid, diphenylthreonedicarboxylic acid, diphenylsulfonedicarboxylic acid, diphenylketonedicarboxylic acid, anthracenedicarboxylic acid, etc.

5 Examples of the aliphatic glycols are polymethylene glycols having 2 to 10 carbon atoms such as ethylene glycol, trimethylene glycol, tetramethylene glycol, pentamethylene glycol, hexamethylene glycol, decamethylene glycol, etc. and alicyclic diols such as cyclohexanedimethanol, etc.

As an aromatic polyester, preferred are those composed mainly of alkylene terephthalate and/or alkylene naphthalate.

10 For the above aromatic polyesters, not more than 20 mol% of the total amount of dicarboxylic acid components may be selected from the above aromatic dicarboxylic acids other than terephthalic acid and/or 2,6-naphthalenedicarboxylic acid, and also may be selected from aliphatic dicarboxylic acids such as adipic acid and sebacic acid and alicyclic dicarboxylic acids such as cyclohexane-1,4-dicarboxylic acid.

Further, not more than 20 mol% of the total amount of glycol components may be selected from the above 15 glycols other than ethylene glycol, and also may be selected from aromatic diols such as hydroquinone, resorcin and 2,2-bis(4-hydroxyphenyl)propane; aliphatic diols having an aromatic ring such as 1,4-dihydroxydimethylbenzene; and polyalkylene glycols (polyoxyalkylene glycols) such as polyethylene glycol, polypropylene glycol and polytetramethylene glycol.

The polyester used in the present invention also includes polyesters in which not more than 20 mol%, based 20 on the total amount of the dicarboxylic acid component and the oxycarboxylic acid component, of a component derived from an oxycarboxylic acid, e.g., an aromatic oxyacid such as hydroxybenzoic acid or an aliphatic oxyacid such as  $\omega$ -hydroxycaproic acid is copolymerized or bonded.

Further, the polyester used in the present invention includes a polyester in which a polycarboxylic acid or a polyhydroxy compound having three or more functional groups such as trimellitic acid, pentaerythritol, etc., 25 is copolymerized in such an amount that the resultant polyester is substantially linear, e.g., not more than 2 mol% based on the total amount of acid components.

As an aromatic polyester in the present invention, particularly preferred are polyethylene terephthalate, polyethylene-2,6-naphthalate and a copolymer composed of a dicarboxylic acid component of which at least 80 30 mol% is terephthalic acid and/or 2,6-dinaphthalenedicarboxylic acid and a glycol component of which at least 80 mol% is ethylene glycol.

The above polyesters are known per se, and can be produced by a known method.

The above polyesters preferably have an inherent viscosity, measured in an *o*-chlorophenol solution at 35°C, of about 0.4 to about 0.9.

The biaxially oriented polyester film of the present invention is a product containing  $\theta$ -aluminum oxide and 35 inert particles having a greater particle diameter than the  $\theta$ -aluminum oxide in any one of the above-described polyesters.

The  $\theta$ -aluminum oxide has an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$ , preferably in the range of from 0.03 to 0.25  $\mu\text{m}$ .

When the above average particle diameter is smaller than 0.02  $\mu\text{m}$ , it is impossible to form fine projections 40 required for scratch resistance and prevention of occurrence of abrasion dust. On the other hand, when the average particle diameter is greater than 0.3  $\mu\text{m}$ , agglomerates of primary particles are too large, and abrasion dust is liable to occur.

The  $\theta$ -aluminum oxide has a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , preferably in the range of from 60 to 100  $\text{m}^2/\text{g}$ .

45 When the above specific surface area is less than 50  $\text{m}^2/\text{g}$ , the scratch resistance is improved, but abrasion dust is liable to occur, and further, a metal guide having a well-finished surface is frequently injured by the resultant film. On the other hand, when the specific surface area exceeds 120  $\text{m}^2/\text{g}$ , the particle diameter is too small to form sufficient projections, and the scratch resistance is degraded although the occurrence of abrasion dust is decreased.

50 The  $\theta$ -aluminum oxide further preferably has an entire pore volume in the range of from 0.5 to 1.0  $\text{ml/g}$ .

When the entire pore volume is less than 0.5  $\text{ml/g}$ , the degree of particles being porous is too small, the affinity of the  $\theta$ -aluminum oxide to the polyester decreases, and abrasion dust is liable to occur. When the entire pore volume exceeds 1.0  $\text{ml/g}$ , the particles are fragile and liable to crumble when the film is stretched for biaxial orientation, and it is difficult to form relatively large projections having an effect on scratch resistance against 55 a metal guide having a well-finished surface.

The content of the  $\theta$ -aluminum oxide in the polyester is 0.05 to 1.0 % by weight, preferably 0.1 to 0.6 % by weight.

When the above content is less than 0.05 % by weight, the effect produced by the fine particles contained

decreases. When the above content exceeds 1.0 % by weight, stacking of particles is observed, and abrasion dust is liable to occur.

The biaxially oriented polyester film of the present invention further contains inert particles having larger particle diameters than the  $\theta$ -aluminum oxide.

5 When the above inert particles are absent, the friction coefficient of the film against various guides is high, and not only the film is degraded in resistance to scratch and occurrence of abrasion dust, but also the handling of the film at the film production step is difficult. Further, when the film is used as a base film for magnetic recording media, the film shows poor running properties.

The inert particles can be selected from inert inorganic particles and inert heat-resistant polymer particles.

10 Examples of the material for the inert inorganic particles are silicon dioxide, titanium oxide, talc, kaolin, calcium carbonate and  $\alpha$ —,  $\theta$ —,  $\delta$ —,  $\gamma$ —,  $\eta$ —,  $\rho$ —,  $\kappa$ — or  $\chi$ —aluminum oxide.

Examples of the inert heat-resistant polymer particles are silicone resin particles, crosslinked acrylic particles, crosslinked polystyrene resin particles, fluorine resin particles and polyimide particles.

The above inert particles may be used alone or in combination of two or more.

15 The inert particles preferably have an average particle diameter in the range of from 0.2 to 1.2  $\mu\text{m}$ .

The inert particles are required to have a particle diameter greater than the  $\theta$ -aluminum oxide having an average particle diameter of 0.02 to 0.3  $\mu\text{m}$ . The content of the inert particles in the polyester is 0.01 to 1.5 % by weight, preferably 0.05 to 1.5 % by weight, more preferably 0.05 to 1.0 % by weight.

20 The  $\theta$ -aluminum oxide and the inert particles may be incorporated and dispersed in the polyester by a conventional method known in the field of this art.

The biaxially oriented polyester film of the present invention can be produced, for example, by melt-extruding a polyester at a temperature ranging from the melting point of the polyester ( $T_m$ : °C) to  $(T_m + 70)^\circ\text{C}$  to obtain an unstretched film having an inherent viscosity of 0.35 to 0.9 dl/g, stretching monoaxially (in the width or length direction) the unstretched film at a stretch ratio of 2.5 to 5.0 at a temperature between  $(T_g - 10)^\circ\text{C}$  and  $(T_g + 70)^\circ\text{C}$  ( $T_g$  stands for the glass transition temperature of the polyester), and then stretching the resultant film at right angles with the above stretching direction (e.g., in the width direction when the unstretched film had been first stretched in the length direction) at a stretch ratio of 2.5 to 5.0 at a temperature between  $T_g$  (°C) and  $(T_g + 70)^\circ\text{C}$ . In this case, the stretch ratio in terms of an area is 9 to 22, preferably 12 to 22. The biaxial stretching may be carried out at the same time or consecutively.

30 Further, the resultant biaxially oriented polyester film may be thermally set at a temperature between  $(T_g + 70)^\circ\text{C}$  and  $T_m$  (°C). For example, it is preferred that the polyethylene terephthalate film be thermally set at a temperature between 190° C and 230° C. The time for the thermal setting is, for example, 1 to 60 seconds.

The biaxially oriented polyester film of the present invention preferably has, on the film surface, projections of which the size, number and distribution are suitable for achievement of the objects of the present invention.

35 For this purpose, the projections satisfy the following relationship,

$$-11.4x + 4 < \log y < -10.0x + 5$$

in which  $x$  is a distance ( $\mu\text{m}$ ) in the height direction from a standard level and  $y$  is a number of projections (pieces/ $\text{mm}^2$ ) counted when the projections are cut at a height of  $x$ ,

provided that  $x$  is in the range of not less than 0.05  $\mu\text{m}$  and that  $y$  is in the range of not less than 30/ $\text{mm}^2$ .

40 The biaxially oriented polyester film of the present invention preferably has a running friction coefficient ( $\mu\text{k}$ ) of not more than 0.3.

The biaxially oriented polyester film of the present invention also preferably has a running friction coefficient increment ( $\Delta \mu\text{k}$ ) of not more than 0.15.

45 The biaxially oriented polyester film of the present invention has a thickness, for example, in the range of from 4 to 100  $\mu\text{m}$ .

Having excellent scratch resistance and abrasion resistance against any of a metal guide having a well-finished surface, a metal guide having an insufficiently finished surface and a plastic guide, the biaxially oriented polyester film of the present invention is very useful as a substrate film for magnetic recording media.

50 According to the present invention, there is therefore provided a magnetic recording medium comprising the above biaxially oriented polyester film of the present invention and a magnetic layer formed thereon.

Further, a study of the present inventors has showed that the above objects of the present invention can be similarly achieved also by the use of mixed ( $\alpha + \gamma$ ) aluminum oxide in place of the  $\theta$ -aluminum oxide.

According to the present invention, therefore, there is also provided a biaxially oriented polyester film for magnetic recording media, which comprises:

- 55 (A) 0.05 to 1.0 % by weight of mixed ( $\alpha + \gamma$ ) aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and  
(B) 0.01 to 1.5 % by weight of inert particles having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the mixed ( $\alpha + \gamma$ ) aluminum oxide.

In the above biaxially oriented polyester film, the above mixed ( $\alpha + \gamma$ ) aluminum oxide means aluminum oxide in which each particle includes an  $\alpha$ -type crystal form and a  $\gamma$ -type crystal form, and it does not mean a mixture of  $\alpha$ -type particles and  $\gamma$ -type particles.

The mixed ( $\alpha + \gamma$ ) aluminum oxide preferably has 3 to 30 % by weight of an  $\alpha$ -type crystal form and 70 to 97 % by weight of a  $\gamma$ -type crystal form. More preferably, it has 10 to 25 % by weight of an  $\alpha$ -type crystal form and 75 to 90 % by weight of a  $\gamma$ -type crystal form.

Similarly to the above  $\theta$ -aluminum oxide, the mixed ( $\alpha + \gamma$ ) aluminum oxide has an average particle diameter of 0.02 to 0.3  $\mu$ m, preferably 0.03 to 0.25  $\mu$ m, and a specific surface area of 50 to 120 m<sup>2</sup>/g, preferably 60 to 100 m<sup>2</sup>/g.

The mixed ( $\alpha + \gamma$ ) aluminum oxide also preferably has an entire pore volume in the range of from 0.5 to 1.0 ml/g.

It should be understood that the previous description of the biaxially oriented polyester film using  $\theta$ -aluminum oxide can be applied to the above biaxially oriented polyester film directly or by replacing the  $\theta$ -aluminum oxide with the mixed ( $\alpha + \gamma$ ) aluminum oxide, except for the above description in regard to the mixed ( $\alpha + \gamma$ ) aluminum oxide.

The biaxially oriented polyester film using the mixed ( $\alpha + \gamma$ ) aluminum oxide, provided by the present invention, is also naturally very useful as a film for magnetic recording media. Therefore, the present invention also provides a magnetic recording medium comprising this biaxially oriented polyester film and a magnetic layer formed thereon.

In the production of a magnetic recording medium, a magnetic recording layer may be formed on the biaxially oriented polyester film of the present invention as a substrate by a method in which, for example, a magnetic coating composition is prepared by kneading a magnetic powder such as ferromagnetic iron oxide (e.g.,  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> or Co-containing  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>) or a ferromagnetic metal powder (e.g., pure Fe and an iron alloy such as Fe-Ni or Fe-Ni-Co) with a binder, and coated on the biaxially oriented polyester film (nonmagnetic support). The biaxially oriented polyester film of the present invention is particularly suitable for use in magnet recording media of high quality containing a ferromagnetic iron oxide powder.

Magnetic recording media having a polyester film as a substrate have found extensive use, for example, in video tapes, audio tapes, computer tapes and floppy discs. In such applications, there has been an increasing demand for high density recording and small sizes, and accordingly, the polyester film as a substrate has been strongly required to have a flat surface, lubricity, excellent handability and a small thickness.

The present invention will be further detailed by reference to Examples.

The physical property values and characteristics in the present specification including Examples were measured or defined as follows.

#### (1) Average particle diameter (DP):

Particles were measured for diameters with a centrifugal particle size analyzer (model CP-50 supplied by Shimadzu Corporation), and there was prepared a cumulative curve of particles of individual diameters and their amounts calculated on the basis of the resultant centrifugal sedimentation curve. In the cumulative curve, a particle diameter corresponding to a 50 mass percent was read, and the particle diameter value read was defined as an average particle diameter of the particles (see "Particle Size Measuring Technique", pages 242-247, 1975, issued by Nikkan Kogyo Press).

#### (2) Specific surface area and Entire pore volume:

Particles were measured for a specific surface area and an entire pore volume according to a BET method with Autosorb-1 supplied by Quantachrome Corp.

#### (3) Running friction coefficient ( $\mu$ k) of film:

A film was measured for a running friction coefficient as follows, using an apparatus shown in Fig. 1.

In Fig. 1, numeral 1 indicates an unwinding reel, numeral 2 indicates a tension controller, numerals 3, 5, 6, 8, 9 and 11 indicate free rollers, numeral 4 indicates a tension detector (inlet), numeral 7 indicates a fixed rod made of stainless steel SUS304 (outer diameter 5 mm $\phi$ , surface roughness Ra = 0.02  $\mu$ m), numeral 10 indicates a tension detector (outlet), numeral 12 is a guide roller, and numeral 13 indicates a take-up reel.

Under an environment having a temperature of 20° C and a humidity of 60 %, a film cut to a width of 1/2 inch was allowed to run (frictionally) at a rate of 200 cm/minute in a contact with the fixed rod at an angle  $\theta = (152/180)\pi$  radian (152°). When a tension T<sub>1</sub> at the inlet was adjusted to 35 g with the tension controller 2, and

after the film ran 90 m, an outlet tension ( $T_2$ : g) was detected with the outlet tension detector, and the running friction coefficient ( $\mu_k$ ) was calculated on the basis of the following equation.

$$\mu_k = (2.303/\theta) \log(T_2/T_1) = 0.868 \log(T_2/35)$$

5 (4) Surface roughness:

A film was measured for an Ra (centerline average roughness) according to JIS B 0601.

10 A chart (film surface roughness curve) was drawn with a needle-contacting type surface roughness tester (SURFCOM 3B supplied by Tokyo Seimitsu Co., Ltd.) with a needle radius of 2  $\mu\text{m}$  under a load of 0.07 g. A portion having a measured length L was taken from the film surface roughness curve in the direction of its center line. The center line of this portion taken was assumed to be X axis and the direction of the longitudinal multiplication, Y axis, and the roughness curve was expressed as  $Y = f(x)$ . The value (Ra;  $\mu\text{m}$ ) given by the following equation was defined as a surface roughness.

$$15 \quad Ra = \frac{1}{L} \int_0^L |f(x)| dx$$

20 In the present invention, the measuring length was set at 1.25 mm, and the measurement was made on 8 portions having the measuring length. The three largest measured values were excluded, and Ra was expressed in terms of an average of the remaining five measured values.

(5) Scratch resistance and abrasion resistance:

25

In the same apparatus as that shown in Fig. 1 and used for the measurement of the running friction coefficient  $\mu_k$ , a film was allowed to run 200 m at a contact angle of 30° at a rate of 300 m/minute and at an inlet tension of 50 g. After the film was allowed to run as above, the film was evaluated on abrasion dust adhering to the fixed rod 7 and scratch occurred on the film surface.

30

In the above test, the following fixed rods were used:

Method A: a 6  $\phi$  film guide made of stainless steel SUS 304 and having a well-finished surface (surface roughness Ra = 0.015  $\mu\text{m}$ ).

Method B: a 6  $\phi$  film guide prepared by cylindrically bending a SUS sintered plate and having a insufficiently finished surface (surface roughness Ra = 0.15  $\mu\text{m}$ )

35

Method C: a 6  $\phi$  film guide formed of a carbon black-containing polyacetal.

<Evaluation on abrasion dust>

40

- A: No abrasion dust was found.
- B: Abrasion dust was slightly found.
- C: Abrasion dust was clearly found.
- D: Abrasion dust heavily adhered.

<Evaluation on scratch>

45

- A: No scratch was found.
- B: 1 to 5 scratches were found.
- C: 6 to 15 scratches were found.
- D: 16 or more scratches were found.

50

(6) Scratch resistance at a low-rate repetition:

In the same apparatus as that shown in Fig. 1 and used for the measurement of the running friction coefficient  $\mu_k$ , a film was allowed to run on the fixed rod in a running length of 10 m reciprocally 50 times. After the film was allowed to run as above, the film was evaluated on scratch resistance.

55

In the above test, the following fixed rods were used:

Method A: a 6  $\phi$  film guide made of SUS 304 and having a well-finished surface (surface roughness Ra = 0.015  $\mu\text{m}$ ).

Method B: a 6  $\phi$  film guide prepared by cylindrically bending a SUS sintered plate and having a insufficiently finished surface (surface roughness  $R_a = 0.15 \mu\text{m}$ )

Method C: a 6  $\phi$  film guide formed of a carbon black-containing polyacetal.

5 <Evaluation on scratch>

A: No scratch was found.

B: 1 to 5 scratches were found.

C: 6 to 15 scratches were found.

10 D: 16 or more scratches were found.

(7) Running friction coefficient increment ( $\Delta\mu_k$ ) of film:

In the same apparatus as that shown in Fig. 1 and used for the measurement of the running friction coefficient  $\mu_k$ , a film having a length of 10 m was allowed to run at a rate of 2 m/minute repetitively 50 times. The friction coefficient when the film was allowed to run for the first time was taken as  $\mu_{k1}$ , and the friction coefficient when it was allowed to run for the fiftieth time was taken as  $\mu_{k50}$ . And the running friction coefficient increment  $\Delta\mu_k$  was calculated by the following equation.

$$\Delta\mu_k = \mu_{k50} - \mu_{k1}.$$

20

(8) Method of measurement of projection distribution:

A three-dimensional (stereoscopic) image of a profile of projections on a film surface was formed with a three-dimensional roughness tester (SE-30K, supplied by Kosaka Laboratory Ltd.) under the conditions where the needle diameter was 2  $\mu\text{m}$ R, the needle pressure was 30 mg, the measurement length was 1 mm, the sampling pitch was 2  $\mu\text{m}$ , the cutoff was 0.25 mm, the magnification in the vertical direction was 20,000, the magnification in the horizontal direction was 200, and the scanning line number was 150.

A plane obtained by cutting the above profile with a plane at right angles with the thickness direction of the film so that the total area of cross sections of profiled projections was 70 % of a measured area of the film was taken as a standard level (0 level). The number of projections counted when the profile was cut, in parallel with the plane at the standard level, with a plane apart from the plane at the standard level by a distance of  $x$  ( $\mu\text{m}$ ) in the height direction of the projections was taken as  $y$  (pieces/ $\text{mm}^2$ ). The numbers  $y$  were read by increasing or decreasing  $x$ , and a projection distribution curve was prepared by plotting the numbers read to draw a graph.

35 Examples 1 - 10

Dimethyl terephthalate and ethylene glycol were polymerized according to a conventional method in the presence of manganese acetate as an ester interchange catalyst, antimony trioxide as a polymerization catalyst, phosphorous acid as a stabilizer and additives shown in Table 1 as a lubricant to give polyethylene terephthalate having an inherent viscosity of 0.62 (in o-chlorophenol, at 35° C).

The pellets of the above polyethylene terephthalate were dried at 170° C for 3 hours, and then fed to an extruder through a hopper. The pellets were melted at a melting temperature of 280 to 300° C, and the molten polymer was extruded onto a rotary cooling drum having a surface finish of about 0.3s and a surface temperature of 20° C through a die having a slit of 1 mm in the width to give an unstretched film having a thickness of 200  $\mu\text{m}$ .

The above-obtained unstretched film was preliminarily heated to 75° C, and stretched at a stretch ratio of 3.6 between a low-speed roll and a high-speed roll by heating the film with one IR heater having a surface temperature of 900° C and positioned 15 mm above the film. The stretched film was cooled rapidly, fed to a stenter and stretched at a stretch ratio of 3.9 in the width direction at 105° C. The resultant biaxially oriented film was thermally set at a temperature of 205° C for 5 seconds to give a thermally-set biaxially oriented film having a thickness of 14  $\mu\text{m}$ .

Separately, 100 parts by weight (to be abbreviated as "part" hereinafter) of  $\gamma\text{-Fe}_2\text{O}_3$  and the following components were kneaded and dispersed in a ball mill for 12 hours.

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	Polyester polyurethane	12 parts
	Vinyl chloride-vinyl acetate-maleic	
5	anhydride copolymer	10 parts
	$\alpha$ -alumina	5 parts
	Carbon black	1 part
	Butyl acetate	70 parts
10	Methyl ethyl ketone	35 parts
	Cyclohexane	100 parts

After the dispersion, the following components were added:

15	Fatty acid: Oleic acid	1 part
	Fatty acid: Palmitic acid	1 part
20	Fatty acid ester: Amyl stearate	1 part

The resultant mixture was further kneaded for 15 to 30 minutes. Further, 7 parts of a 25 % ethyl acetate solution of a triisocyanate compound was added, and the resultant mixture was dispersed at a high velocity under a shear force to prepare a magnetic coating liquid.

25 The above-obtained magnetic coating liquid was coated on the above 14  $\mu\text{m}$  thick polyester film such that the dry coating was 3.5  $\mu\text{m}$  thick.

The coated polyester film was subjected to an orientation treatment in a direct current magnetic field, and dried at 100° C. The dried coated polyester film was subjected to a calendering treatment, and slit to a width of 1/2 inch to give a magnetic tape having a thickness of 17.5  $\mu\text{m}$ .

30 Table 1 shows the properties of the magnetic tapes obtained in the above manner in each Example.

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Table 1

				Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5
Inert particles	A	Kind		Al <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>
		Specific surface area	m <sup>2</sup> /g	75	85	75	75	75
		Entire pore volume	ml/g	0.75	0.82	0.75	0.75	0.75
		Average particle diameter	μm	0.20	0.21	0.20	0.20	0.20
		Amount added	%	0.3	0.3	0.3	0.3	0.3
		Crystal form		θ form	(α+γ) form	θ form	θ form	θ form
	B	Kind		CaCO <sub>3</sub>	CaCO <sub>3</sub>	CaCO <sub>3</sub>	SiO <sub>2</sub>	kaolin
		Specific surface area	μm	0.6	0.6	0.6	0.6	0.6
		Entire pore volume	%	0.3	0.3	0.15	0.3	0.2
		Kind		-	-	SiO <sub>2</sub>	-	-
		Average particle diameter	μm	-	-	0.6	-	-
		Amount added	%	-	-	0.15	-	-

- to be continued -

Table 1 (continued)

				Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5
Surface roughness Ra			nm	20	20	19	20	22
Running friction coefficient				0.20	0.20	0.17	0.16	0.25
Running friction coefficient increment				0.10	0.10	0.06	0.04	0.15
Projection distribution curve (Fig. 2)				A	-	B	C	D
Scratch resistance and abrasion resistance at high rate	Method A	Abrasion resistance		A	A	B	B	B
		Scratch resistance		A	A	A	A	A
	Method B	Abrasion resistance		A	A	B	B	B
		Scratch resistance		A	A	A	A	A
	Method C	Abrasion resistance		A	A	B	B	A
		Scratch resistance		A	A	A	A	A
Scratch resistance at low-rate repetition	Method A	Scratch resistance		A	A	A	A	B
	Method B	Scratch resistance		A	A	A	A	A
	Method C	Scratch resistance		A	A	A	B	A

As is clearly shown in Table 1, the biaxially oriented polyester film of the present invention not only has excellent lubricity as a substrate film for magnetic recording media but also has excellent abrasion resistance and scratch resistance against all of a metal tap guide having a well-finished surface, a metal tap guide having an insufficiently finished surface and a plastic tap guide. The biaxially oriented polyester film of the present invention has excellent properties as a substrate film for magnetic recording media.

### Claims

1. A biaxially oriented polyester film for magnetic recording media, which comprises:
  - (A) 0.05 to 1.0 % by weight of  $\theta$ -aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and
  - (B) 0.01 to 1.5 % by weight of inert particles having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the  $\theta$ -aluminum oxide.
2. The biaxially oriented polyester film of Claim 1, wherein the  $\theta$ -aluminum oxide has an average particle diameter in the range of from 0.03 to 0.25  $\mu\text{m}$ .
3. The biaxially oriented polyester film of Claim 1, wherein the  $\theta$ -aluminum oxide has a specific surface area in the range of from 60 to 100  $\text{m}^2/\text{g}$ .
4. The biaxially oriented polyester film of Claim 1, wherein the  $\theta$ -aluminum oxide has an entire pore volume in the range of from 0.5 to 1.0  $\text{ml/g}$ .
5. The biaxially oriented polyester film of Claim 1, wherein the inert particles are inert inorganic particles or inert heat-resistant polymer particles.
6. The biaxially oriented polyester film of Claim 5, wherein the inert inorganic particles are formed of a material selected from the group consisting of silicon dioxide, titanium oxide, talc, kaolin, calcium carbonate and  $\alpha$ —,  $\theta$ —,  $\delta$ —,  $\gamma$ —,  $\eta$ —,  $\rho$ —,  $\kappa$ — or  $\chi$ —aluminum oxide.
7. The biaxially oriented polyester film of Claim 5, wherein the inert heat-resistant polymer particles are selected from the group consisting of silicone resin particles, crosslinked acrylic particles, crosslinked polystyrene resin particles, fluorine resin particles and polyimide particles.
8. The biaxially oriented polyester film of Claim 1, wherein the inert particles have an average particle diameter in the range of from 0.2 to 1.2  $\mu\text{m}$ .
9. The biaxially oriented polyester film of Claim 1, wherein the polyester comprises an aromatic dicarboxylic acid as a main acid component and an aliphatic glycol having 2 to 10 carbon atoms as a main glycol component.
10. The biaxially oriented polyester film of Claim 1, which has a running friction coefficient ( $\mu\text{k}$ ) of not more than 0.3.
11. The biaxially oriented polyester film of Claim 1, which has a running friction coefficient increment ( $\Delta\mu\text{k}$ ) of not more than 0.15.
12. The biaxially oriented polyester film of Claim 1, which has projections satisfying the following relationship,
 
$$-11.4x + 4 < \log y < -10.0x + 5$$
 in which  $x$  is a distance ( $\mu\text{m}$ ) in the height direction from a standard level and  $y$  is a number of projections (pieces/ $\text{mm}^2$ ) counted when the projections are cut at a height of  $x$ , provided that  $x$  is in the range of not less than 0.05  $\mu\text{m}$  and that  $y$  is in the range of not less than 30/ $\text{mm}^2$ .
13. The biaxially oriented polyester film of Claim 1, which has a thickness in the range of from 4 to 100  $\mu\text{m}$ .
14. A magnetic recording medium comprising the biaxially oriented polyester film recited in Claim 1 and a magnetic layer formed thereon.

15. A biaxially oriented polyester film for magnetic recording media, which comprises:  
(A) 0.05 to 1.0 % by weight of mixed ( $\alpha + \gamma$ ) aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and  
(B) 0.01 to 1.5 % by weight of inert particles having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the mixed ( $\alpha + \gamma$ ) aluminum oxide.
16. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha + \gamma$ ) aluminum oxide has an average particle diameter in the range of from 0.03 to 0.25  $\mu\text{m}$ .
17. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha + \gamma$ ) aluminum oxide has a specific surface area in the range of from 60 to 100  $\text{m}^2/\text{g}$ .
18. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha + \gamma$ ) aluminum oxide has an entire pore volume in the range of from 0.5 to 1.0  $\text{ml/g}$ .
19. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha + \gamma$ ) aluminum oxide is formed of 3 to 30 % by weight of an  $\alpha$ -type crystal form and 70 to 97 % by weight of a  $\gamma$ -type crystal form.
20. A magnetic recording medium comprising the biaxially oriented polyester film recited in Claim 15 and a magnetic layer formed thereon.

FIG. 1

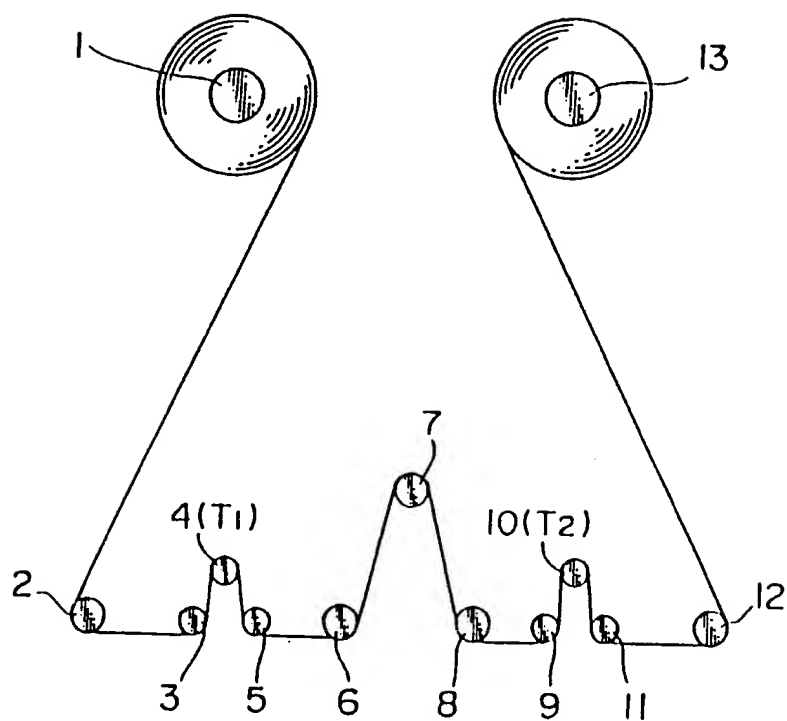
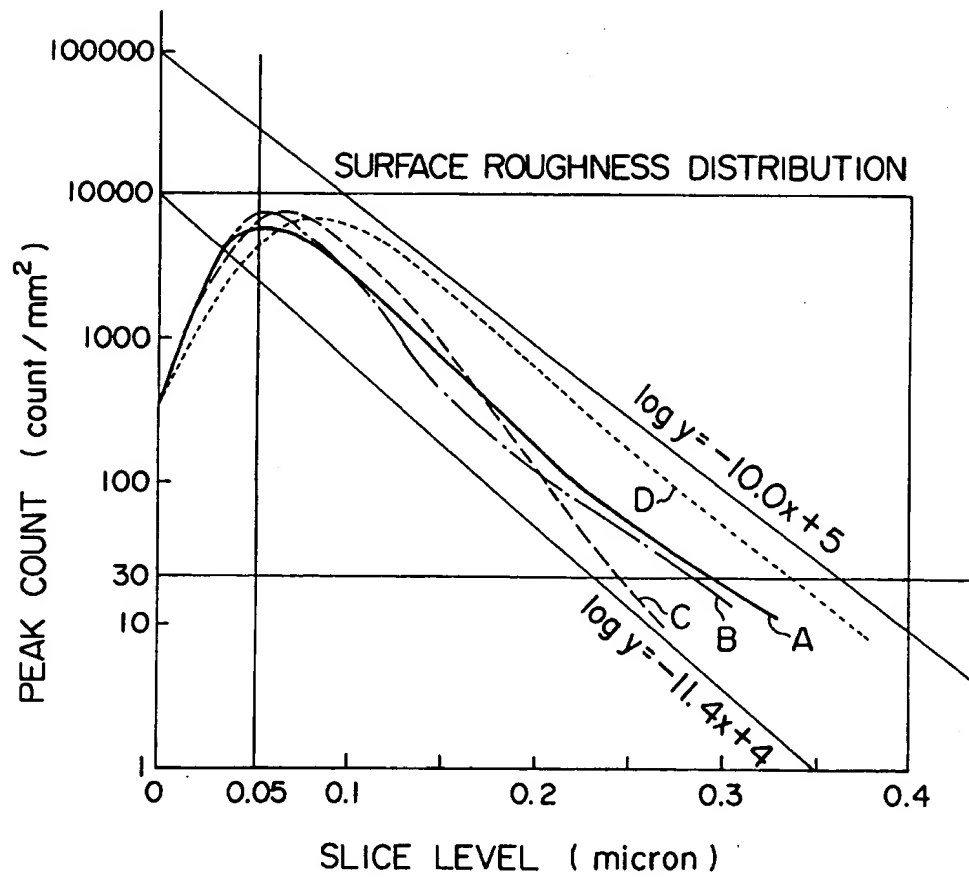


FIG. 2





European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 91311542.4
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
A	DATABASE WPIL, no. 90-111 188 DERWENT PUBLICATIONS LTD., London, GB & JP-A-02 60 937 (DIA FOIL K.K.) 01-03-1990 * Abstract *	1-20	G 11 B 5/704 G 11 B 5/70 G 11 B 5/708
A	DATABASE WPIL, no. 89-188 408 DERWENT PUBLICATIONS LTD., London, GB & JP-A-01-126 340 (TEIJIN K.K.) 18-05-1989 * Abstract *	1-20	
A	EP - A - 0 345 644 (DIAFOIL COMPANY, LTD.) * Abstract *	1-20	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			G 11 B 5/00 C 08 J 5/00 B 29 C 55/00
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 13-03-1992	Examiner BERGER
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons</p> <p>&amp; : member of the same patent family, corresponding document</p>			

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## Description

### Detailed Description of the Invention

5 The present invention relates to a biaxially oriented polyester film for magnetic recording media. More specifically, it relates to a biaxially oriented polyester film for magnetic recording media, which is excellent in a variety of performances required of films for magnetic recording media, particularly abrasion resistance and scratch resistance.

10 Biaxially oriented polyester films typified by a polyethylene terephthalate film are extensively used for magnetic recording media owing to their excellent physical and chemical properties.

In biaxially oriented polyester films, their lubricity and abrasion resistance are crucial factors which influence not only the quality of accomplishment in the manufacturing and processing step but also their product quality.

15 When the biaxially oriented polyester film is poor in the above properties, for example, abrasion dust occurs or the film surface is scratched due to strong friction between a coating roll and the film surface during the formation of a magnetic tape by coating a magnetic layer on a biaxially oriented polyester film. Further, when such a magnetic tape is used as a VTR or data cartridge, abrasion dust or scratch also occurs when the tape is taken into a cassette at a high speed, which might cause drop-out of recorded data.

20 In the recent VTR cassette field, a metal guide of which the surface finish is insufficient or a plastic guide is sometimes used as a guide post fixedly provided in the cassette with the intention of reducing the manufacturing cost. This guide post has a very rough surface. Therefore, in a conventional method for improvement in lubricity and abrasion resistance, e.g., a method in which inorganic particles of silicon oxide, titanium dioxide, calcium carbonate, talc, clay, calcined kaolin, etc., are incorporated (see Japanese Laid-Open Patent Publication No. 57762/1979), or a method in which fine particles containing calcium, lithium or phosphor are precipitated in the polymerization system (see Japanese Patent Publication No. 32914/1977), the resultant magnetic tape provided with no back coating causes abrasion dust or scratch when the tape is taken into a cassette at a high speed, and recorded data is often dropped out.

25 U.S. Patent 5,006,589 discloses a polyester film for magnetic recording media which contains 0.05 to 5 % by weight of inactive inorganic particles (A) having a Mohs hardness of 6 or above and an average particle size of 0.005 to 1.0  $\mu\text{m}$  and 0.01 to 2 % by weight of inactive particles (B) having an average particle size which is greater than that of the particles (A) and in the range of 0.1 to 3.0  $\mu\text{m}$ , the polyester film having an intrinsic viscosity in the range of 0.52 to 0.65.

30 As a material for the above inactive inorganic particles (A),  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiC}$ , zeolite, sellaite,  $\text{TiC}$  and titanium black are disclosed. Further, it is described that  $\text{Al}_2\text{O}_3$  is particularly preferred, and there are disclosed  $\alpha$ —,  $\beta$ —,  $\gamma$ —,  $\kappa$ —,  $\sigma$ —,  $\delta$ —,  $\eta$ —,  $\chi$ — and  $\rho$ —type  $\text{Al}_2\text{O}_3$ 's.

35 The above polyester film shows an effect on the guide posts employed for reducing in the manufacturing cost. However, when a conventional metal guide having a well-finished surface is used, the effect on improvement is insufficient. There is also a method in which inactive particles having a little greater size than the above inactive inorganic particles (A) are used in combination to overcome scratch caused by a metal guide having a well-finished surface. In this method, however, there is observed an adverse influence that abrasion dust newly occurs.

40 It is therefore an object of the present invention to provide a biaxially oriented polyester film for magnetic recording media.

45 It is another object of the present invention to provide a biaxially oriented polyester film for magnetic recording media, which is excellent in abrasion resistance and scratch resistance against not only a conventional metal guide having a well-finished surface but also a recent metal guide having a rough surface and a recent plastic guide, and which is also excellent in lubricity.

The other objects and advantages of the present invention will be apparent from the following description.

50 According to the present invention, the above objects and advantages of the present invention are achieved by a biaxially oriented polyester film for magnetic recording media, which comprises:

(A) 0.05 to 1.0 % by weight of  $\theta$ -aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and

55 (B) 0.01 to 1.5 % by weight of inert particles other than  $\theta$ -aluminum oxide having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the  $\theta$ -aluminum oxide.

Fig. 1 is a schematic view of an apparatus for measuring a running friction coefficient of a biaxially oriented polyester film for magnetic recording media.

Fig. 2 is a projection distribution curve.

The polyester which constitutes the biaxially oriented polyester film of the present invention is preferably selected from aromatic polyesters comprising aromatic dicarboxylic acids as a main acid component and aliphatic glycols having 2 to 10 carbon atoms as a main glycol component. As such a polyester, polyesters which are substantially linear and have film formability in the melt-molding are advantageous.

Examples of the aromatic dicarboxylic acids are terephthalic acid, naphthalenedicarboxylic acid, isophthalic acid, diphenoxyethanedicarboxylic acid, diphenyldicarboxylic acid, diphenyletherdicarboxylic acid, diphenylsulfonedicarboxylic acid, diphenylketonedicarboxylic acid, anthracenedicarboxylic acid, etc.

Examples of the aliphatic glycols are polymethylene glycols having 2 to 10 carbon atoms such as ethylene glycol, trimethylene glycol, tetramethylene glycol, pentamethylene glycol, hexamethylene glycol, decamethylene glycol, etc. and alicyclic diols such as cyclohexanedimethanol, etc.

As an aromatic polyester, preferred are those composed mainly of alkylene terephthalate and/or alkylene naphthalate.

For the above aromatic polyesters, not more than 20 mol% of the total amount of dicarboxylic acid components may be selected from the above aromatic dicarboxylic acids other than terephthalic acid and/or 2,6-naphthalenedicarboxylic acid, and also may be selected from aliphatic dicarboxylic acids such as adipic acid and sebacic acid and alicyclic dicarboxylic acids such as cyclohexane-1,4-dicarboxylic acid.

Further, not more than 20 mol% of the total amount of glycol components may be selected from the above glycols other than ethylene glycol, and also may be selected from aromatic diols such as hydroquinone, resorcin and 2,2-bis(4-hydroxyphenyl)propane; aliphatic diols having an aromatic ring such as 1,4-dihydroxydimethylbenzene; and polyalkylene glycols (polyoxyalkylene glycols) such as polyethylene glycol, polypropylene glycol and polytetramethylene glycol.

The polyester used in the present invention also includes polyesters in which not more than 20 mol%, based on the total amount of the dicarboxylic acid component and the oxycarboxylic acid component, of a component derived from an oxycarboxylic acid, e.g., an aromatic oxyacid such as hydroxybenzoic acid or an aliphatic oxyacid such as  $\omega$ -hydroxycaproic acid is copolymerized or bonded.

Further, the polyester used in the present invention includes a polyester in which a polycarboxylic acid or a polyhydroxy compound having three or more functional groups such as trimellitic acid, pentaerythritol, etc., is copolymerized in such an amount that the resultant polyester is substantially linear, e.g., not more than 2 mol% based on the total amount of acid components.

As an aromatic polyester in the present invention, particularly preferred are polyethylene terephthalate, polyethylene-2,6-naphthalate and a copolymer composed of a dicarboxylic acid component of which at least 80 mol% is terephthalic acid and/or 2,6-dinaphthalenedicarboxylic acid and a glycol component of which at least 80 mol% is ethylene glycol.

The above polyesters are known per se, and can be produced by a known method.

The above polyesters preferably have an inherent viscosity, measured in an *o*-chlorophenol solution at 35°C, of about 0.4 to about 0.9.

The biaxially oriented polyester film of the present invention is a product containing  $\theta$ -aluminum oxide and inert particles having a greater particle diameter than the  $\theta$ -aluminum oxide in any one of the above-described polyesters.

The  $\theta$ -aluminum oxide has an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$ , preferably in the range of from 0.03 to 0.25  $\mu\text{m}$ .

When the above average particle diameter is smaller than 0.02  $\mu\text{m}$ , it is impossible to form fine projections required for scratch resistance and prevention of occurrence of abrasion dust. On the other hand, when the average particle diameter is greater than 0.3  $\mu\text{m}$ , agglomerates of primary particles are too large, and abrasion dust is liable to occur.

The  $\theta$ -aluminum oxide has a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , preferably in the range of from 60 to 100  $\text{m}^2/\text{g}$ .

When the above specific surface area is less than 50  $\text{m}^2/\text{g}$ , the scratch resistance is improved, but abrasion dust is liable to occur, and further, a metal guide having a well-finished surface is frequently injured by the resultant film. On the other hand, when the specific surface area exceeds 120  $\text{m}^2/\text{g}$ , the particle diameter is too small to form sufficient projections, and the scratch resistance is degraded although the occurrence of abrasion dust is decreased.

The  $\theta$ -aluminum oxide further preferably has an entire pore volume in the range of from 0.5 to 1.0  $\text{ml/g}$ .

When the entire pore volume is less than 0.5  $\text{ml/g}$ , the degree of particles being porous is too small, the affinity of the  $\theta$ -aluminum oxide to the polyester decreases, and abrasion dust is liable to occur. When the entire pore volume exceeds 1.0  $\text{ml/g}$ , the particles are fragile and liable to crumble when the film is stretched for biaxial orientation, and it is difficult to form relatively large projections having an effect on scratch resistance against a metal guide having a well-finished surface.

The content of the  $\theta$ -aluminum oxide in the polyester is 0.05 to 1.0 % by weight, preferably 0.1 to 0.6 % by weight.

When the above content is less than 0.05 % by weight, the effect produced by the fine particles contained decreases. When the above content exceeds 1.0 % by weight, stacking of particles is observed, and abrasion dust is liable to occur.

The biaxially oriented polyester film of the present invention further contains inert particles having larger particle diameters than the  $\theta$ -aluminum oxide.

When the above inert particles are absent, the friction coefficient of the film against various guides is high, and not only the film is degraded in resistance to scratch and occurrence of abrasion dust, but also the handling of the film at the film production step is difficult. Further, when the film is used as a base film for magnetic recording media, the film shows poor running properties.

The inert particles can be selected from inert inorganic particles and inert heat-resistant polymer particles.

Examples of the material for the inert inorganic particles are silicon dioxide, titanium oxide, talc, kaolin, calcium carbonate and  $\alpha$ -,  $\delta$ -,  $\gamma$ -,  $\eta$ -,  $\rho$ -,  $\kappa$ - or  $\chi$ -aluminum oxide.

Examples of the inert heat-resistant polymer particles are silicone resin particles, crosslinked acrylic particles, crosslinked polystyrene resin particles, fluorine resin particles and polyimide particles.

The above inert particles may be used alone or in combination of two or more.

The inert particles preferably have an average particle diameter in the range of from 0.2 to 1.2  $\mu\text{m}$ .

The inert particles are required to have a particle diameter greater than the  $\theta$ -aluminum oxide having an average particle diameter of 0.02 to 0.3  $\mu\text{m}$ . The content of the inert particles in the polyester is 0.01 to 1.5 % by weight, preferably 0.05 to 1.5 % by weight, more preferably 0.05 to 1.0 % by weight.

The  $\theta$ -aluminum oxide and the inert particles may be incorporated and dispersed in the polyester by a conventional method known in the field of this art.

The biaxially oriented polyester film of the present invention can be produced, for example, by melt-extruding a polyester at a temperature ranging from the melting point of the polyester ( $T_m$ : °C) to ( $T_m + 70$ )°C to obtain an unstretched film having an inherent viscosity of 0.35 to 0.9 dl/g, stretching monoaxially (in the width or length direction) the unstretched film at a stretch ratio of 2.5 to 5.0 at a temperature between ( $T_g - 10$ )°C and ( $T_g + 70$ )°C ( $T_g$  stands for the glass transition temperature of the polyester), and then stretching the resultant film at right angles with the above stretching direction (e.g., in the width direction when the unstretched film had been first stretched in the length direction) at a stretch ratio of 2.5 to 5.0 at a temperature between  $T_g$  (°C) and ( $T_g + 70$ )°C. In this case, the stretch ratio in terms of an area is 9 to 22, preferably 12 to 22. The biaxial stretching may be carried out at the same time or consecutively.

Further, the resultant biaxially oriented polyester film may be thermally set at a temperature between ( $T_g + 70$ )°C and  $T_m$  (°C). For example, it is preferred that the polyethylene terephthalate film be thermally set at a temperature between 190° C and 230° C. The time for the thermal setting is, for example, 1 to 60 seconds.

The biaxially oriented polyester film of the present invention preferably has, on the film surface, projections of which the size, number and distribution are suitable for achievement of the objects of the present invention. For this purpose, the projections satisfy the following relationship,

$$-11.4x + 4 < \log y < -10.0x + 5$$

in which  $x$  is a distance ( $\mu\text{m}$ ) in the height direction from a standard level and  $y$  is a number of projections (pieces/ $\text{mm}^2$ ) counted when the projections are cut at a height of  $x$ , provided that  $x$  is in the range of not less than 0.05  $\mu\text{m}$  and that  $y$  is in the range of not less than 30/ $\text{mm}^2$ .

The biaxially oriented polyester film of the present invention preferably has a running friction coefficient ( $\mu\text{k}$ ) of not more than 0.3.

The biaxially oriented polyester film of the present invention also preferably has a running friction coefficient increment ( $\Delta \mu\text{k}$ ) of not more than 0.15.

The biaxially oriented polyester film of the present invention has a thickness, for example, in the range of from 4 to 100  $\mu\text{m}$ .

Having excellent scratch resistance and abrasion resistance against any of a metal guide having a well-finished surface, a metal guide having an insufficiently finished surface and a plastic guide, the biaxially oriented polyester film of the present invention is very useful as a substrate film for magnetic recording media.

According to the present invention, there is therefore provided a magnetic recording medium comprising the above biaxially oriented polyester film of the present invention and a magnetic layer formed thereon.

Further, a study of the present inventors has showed that the above objects of the present invention can be similarly achieved also by the use of mixed ( $\alpha + \gamma$ ) aluminum oxide in place of the  $\theta$ -aluminum oxide.

According to the present invention, therefore, there is also provided a biaxially oriented polyester film for magnetic recording media, which comprises:

(A) 0.05 to 1.0 % by weight of mixed ( $\alpha + \gamma$ ) aluminum oxide having an average particle diameter in the

range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and (B) 0.01 to 1.5 % by weight of, in rt particles other than mixed ( $\alpha + \gamma$ ) aluminium oxide having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the mixed ( $\alpha + \gamma$ ) aluminium oxide.

5 In the above biaxially oriented polyester film, the above mixed ( $\alpha + \gamma$ ) aluminum oxide means aluminum oxide in which each particle includes an  $\alpha$ -type crystal form and a  $\gamma$ -type crystal form, and it does not mean a mixture of  $\alpha$ -type particles and  $\gamma$ -type particles.

The mixed ( $\alpha + \gamma$ ) aluminum oxide preferably has 3 to 30 % by weight of an  $\alpha$ -type crystal form and 70 to 97 % by weight of a  $\gamma$ -type crystal form. More preferably, it has 10 to 25 % by weight of an  $\alpha$ -type crystal form and 75 to 90 % by weight of a  $\gamma$ -type crystal form.

10 Similarly to the above  $\theta$ -aluminum oxide, the mixed ( $\alpha + \gamma$ ) aluminum oxide has an average particle diameter of 0.02 to 0.3  $\mu\text{m}$ , preferably 0.03 to 0.25  $\mu\text{m}$ , and a specific surface area of 50 to 120  $\text{m}^2$ , preferably 60 to 100  $\text{m}^2/\text{g}$ .

The mixed ( $\alpha + \gamma$ ) aluminum oxide also preferably has an entire pore volume in the range of from 0.5 to 1.0  $\text{ml/g}$ .

15 It should be understood that the previous description of the biaxially oriented polyester film using  $\theta$ -aluminum oxide can be applied to the above biaxially oriented polyester film directly or by replacing the  $\theta$ -aluminum oxide with the mixed ( $\alpha + \gamma$ ) aluminum oxide, except for the above description in regard to the mixed ( $\alpha + \gamma$ ) aluminum oxide.

20 The biaxially oriented polyester film using the mixed ( $\alpha + \gamma$ ) aluminum oxide, provided by the present invention, is also naturally very useful as a film for magnetic recording media. Therefore, the present invention also provides a magnetic recording medium comprising this biaxially oriented polyester film and a magnetic layer formed thereon.

In the production of a magnetic recording medium, a magnetic recording layer may be formed on the biaxially oriented polyester film of the present invention as a substrate by a method in which, for example, a magnetic coating composition is prepared by kneading a magnetic powder such as ferromagnetic iron oxide (e.g.,  $\gamma\text{-Fe}_2\text{O}_3$  or Co-containing  $\gamma\text{-Fe}_2\text{O}_3$ ) or a ferromagnetic metal powder (e.g., pure Fe and an iron alloy such as Fe-Ni or Fe-Ni-Co) with a binder, and coated on the biaxially oriented polyester film (nonmagnetic support). The biaxially oriented polyester film of the present invention is particularly suitable for use in magnetic recording media of high quality containing a ferromagnetic iron oxide powder.

Magnetic recording media having a polyester film as a substrate have found extensive use, for example, in video tapes, audio tapes, computer tapes and floppy discs. In such applications, there has been an increasing demand for high density recording and small sizes, and accordingly, the polyester film as a substrate has been strongly required to have a flat surface, lubricity, excellent handlability and a small thickness.

35 The present invention will be further detailed by reference to Examples.

The physical property values and characteristics in the present specification including Examples were measured or defined as follows.

(1) Average particle diameter (DP):

40 Particles were measured for diameters with a centrifugal particle size analyzer (model CP-50 supplied by Shimadzu Corporation), and there was prepared a cumulative curve of particles of individual diameters and their amounts calculated on the basis of the resultant centrifugal sedimentation curve. In the cumulative curve, a particle diameter corresponding to a 50 mass percent was read, and the particle diameter value read was defined as an average particle diameter of the particles (see "Particle Size Measuring Technique", pages 242-247, 1975, issued by Nikkan Kogyo Press).

45 (2) Specific surface area and Entire pore volume:

Particles were measured for a specific surface area and an entire pore volume according to a BET method with Autosorb-1 supplied by Quantachrome Corp.

(3) Running friction coefficient ( $\mu\text{k}$ ) of film:

A film was measured for a running friction coefficient as follows, using an apparatus shown in Fig. 1.

50 In Fig. 1, numeral 1 indicates an unwinding reel, numeral 2 indicates a tension controller, numerals 3, 5, 6, 8, 9 and 11 indicate free rollers, numeral 4 indicates a tension detector (inlet), numeral 7 indicates a fixed rod made of stainless steel SUS304 (outer diameter 5 mm $\phi$ , surface roughness  $R_a = 0.02 \mu\text{m}$ ), numeral 10 indicates a tension detector (outlet), numeral 12 is a guide roller, and numeral 13 indicates a take-up reel.

55 Under an environment having a temperature of 20° C and a humidity of 60 %, a film cut to a width of 1/2 inch was allowed to run (frictionally) at a rate of 200 cm/minute in a contact with the fixed rod at an angle  $\theta = (152/180)\pi$  radian (152°). When a tension  $T_1$  at the inlet was adjusted to 35 g with the tension controller 2, and after the film ran 90 m, an outlet tension ( $T_2$ : g) was detected with the outlet tension d -

tector, and the running friction coefficient ( $\mu_k$ ) was calculated on the basis of the following equation.

$$\mu_k = (2.303/\theta) \log(T_2/T_1) = 0.868 \log(T_2/35)$$

(4) Surface roughness:

A film was measured for an  $R_a$  (centerline average roughness) according to JIS B 0601.

A chart (film surface roughness curve) was drawn with a needle-contacting type surface roughness tester (SURFCOM 3B supplied by Tokyo Seimitsu Co., Ltd.) with a needle radius of 2  $\mu\text{m}$  under a load of 0.07 g. A portion having a measured length  $L$  was taken from the film surface roughness curve in the direction of its center line. The center line of this portion taken was assumed to be X axis and the direction of the longitudinal multiplication, Y axis, and the roughness curve was expressed as  $Y = f(x)$ . The value ( $R_a$ ;  $\mu\text{m}$ ) given by the following equation was defined as a surface roughness.

$$R_a = \frac{1}{L} \int_0^L |f(x)| dx$$

In the present invention, the measuring length was set at 1.25 mm, and the measurement was made on 8 portions having the measuring length. The three largest measured values were excluded, and  $R_a$  was expressed in terms of an average of the remaining five measured values.

(5) Scratch resistance and abrasion resistance:

In the same apparatus as that shown in Fig. 1 and used for the measurement of the running friction coefficient  $\mu_k$ , a film was allowed to run 200 m at a contact angle of 30° at a rate of 300 m/minute and at an inlet tension of 50 g. After the film was allowed to run as above, the film was evaluated on abrasion dust adhering to the fixed rod 7 and scratch occurred on the film surface.

In the above test, the following fixed rods were used:

Method A: a 6  $\phi$  film guide made of stainless steel SUS 304 and having a well-finished surface (surface roughness  $R_a = 0.015 \mu\text{m}$ ).

Method B: a 6  $\phi$  film guide prepared by cylindrically bending a SUS sintered plate and having a insufficiently finished surface (surface roughness  $R_a = 0.15 \mu\text{m}$ )

Method C: a 6  $\phi$  film guide formed of a carbon black-containing polyacetal.

<Evaluation on abrasion dust>

A: No abrasion dust was found.

B: Abrasion dust was slightly found.

C: Abrasion dust was clearly found.

D: Abrasion dust heavily adhered.

<Evaluation on scratch>

A: No scratch was found.

B: 1 to 5 scratches were found.

C: 6 to 15 scratches were found.

D: 16 or more scratches were found.

(6) Scratch resistance at a low-rate repetition:

In the same apparatus as that shown in Fig. 1 and used for the measurement of the running friction coefficient  $\mu_k$ , a film was allowed to run on the fixed rod in a running length of 10 m reciprocally 50 times. After the film was allowed to run as above, the film was evaluated on scratch resistance.

In the above test, the following fixed rods were used:

Method A: a 6  $\phi$  film guide made of SUS 304 and having a well-finished surface (surface roughness  $R_a = 0.015 \mu\text{m}$ ).

Method B: a 6  $\phi$  film guide prepared by cylindrically bending a SUS sintered plate and having a insufficiently finished surface (surface roughness  $R_a = 0.15 \mu\text{m}$ )

Method C: a 6  $\phi$  film guide formed of a carbon black-containing polyacetal.

<Evaluation on scratch>

A: No scratch was found.

B: 1 to 5 scratches were found.

C: 6 to 15 scratches were found.

D: 16 or more scratches were found.

(7) Running friction coefficient increment ( $\Delta\mu_k$ ) of film:

In the same apparatus as that shown in Fig. 1 and used for the measurement of the running friction coefficient  $\mu_k$ , a film having a length of 10 m was allowed to run at a rate of 2 m/minute repetitively 50 times. The friction coefficient when the film was allowed to run for the first time was taken as  $\mu_{k1}$ , and

the friction coefficient when it was allowed to run for the fiftieth time was taken as  $\mu_{k_{50}}$ . And the running friction coefficient increment  $\Delta\mu_k$  was calculated by the following equation.

$$\Delta\mu_k = \mu_{k_{50}} - \mu_{k_1}$$

(8) Method of measurement of projection distribution:

5 A three-dimensional (stereoscopic) image of a profile of projections on a film surface was formed with a three-dimensional roughness tester (SE-30K, supplied by Kosaka Laboratory Ltd.) under the conditions where the needle diameter was 2  $\mu\text{m}$ , the needle pressure was 30 mg, the measurement length was 1 mm, the sampling pitch was 2  $\mu\text{m}$ , the cutoff was 0.25 mm, the magnification in the vertical direction was 20,000, the magnification in the horizontal direction was 200, and the scanning line number was 150.

10 A plane obtained by cutting the above profile with a plane at right angles with the thickness direction of the film so that the total area of cross sections of profiled projections was 70 % of a measured area of the film was taken as a standard level (0 level). The number of projections counted when the profile was cut, in parallel with the plane at the standard level, with a plane apart from the plane at the standard level by a distance of x ( $\mu\text{m}$ ) in the height direction of the projections was taken as y (pieces/ $\text{mm}^2$ ). The numbers y were read by increasing or decreasing x, and a projection distribution curve was prepared by plotting the numbers read to draw a graph.

#### Examples 1 - 10

20 Dimethyl terephthalate and ethylene glycol were polymerized according to a conventional method in the presence of manganese acetate as an ester interchange catalyst, antimony trioxide as a polymerization catalyst, phosphorous acid as a stabilizer and additives shown in Table 1 as a lubricant to give polyethylene terephthalate having an inherent viscosity of 0.62 (in o-chlorophenol, at 35° C).

25 The pellets of the above polyethylene terephthalate were dried at 170° C for 3 hours, and then fed to an extruder through a hopper. The pellets were melted at a melting temperature of 280 to 300° C, and the molten polymer was extruded onto a rotary cooling drum having a surface finish of about 0.3s and a surface temperature of 20° C through a die having a slit of 1 mm in the width to give an unstretched film having a thickness of 200  $\mu\text{m}$ .

30 The above-obtained unstretched film was preliminarily heated to 75° C, and stretched at a stretch ratio of 3.6 between a low-speed roll and a high-speed roll by heating the film with one IR heater having a surface temperature of 900° C and positioned 15 mm above the film. The stretched film was cooled rapidly, fed to a stenter and stretched at a stretch ratio of 3.9 in the width direction at 105° C. The resultant biaxially oriented film was thermally set at a temperature of 205° C for 5 seconds to give a thermally-set biaxially oriented film having a thickness of 14  $\mu\text{m}$ .

35 Separately, 100 parts by weight (to be abbreviated as "part" hereinafter) of  $\gamma\text{-Fe}_2\text{O}_3$  and the following components were kneaded and dispersed in a ball mill for 12 hours.

	Polyester polyurethane	12 parts
40	Vinyl chloride-vinyl acetate-maleic anhydride copolymer	10 parts
	$\alpha$ -alumina	5 parts
	Carbon black	1 part
45	Butyl acetate	70 parts
	Methyl ethyl ketone	35 parts
	Cyclohexane	100 parts

50

After the dispersion, the following components were added:

	Fatty acid: Oleic acid	1 part
55	Fatty acid: Palmitic acid	1 part
	Fatty acid ester: Amyl stearate	1 part

The resultant mixture was further kneaded for 15 to 30 minutes. Further, 7 parts of a 25 % ethyl acetate solution of a triisocyanate compound was added, and the resultant mixture was dispersed at a high velocity under a shear force to prepare a magnetic coating liquid.

The above-obtained magnetic coating liquid was coated on the above 14  $\mu\text{m}$  thick polyester film such that the dry coating was 3.5  $\mu\text{m}$  thick.

The coated polyester film was subjected to an orientation treatment in a direct current magnetic field, and dried at 100° C. The dried coated polyester film was subjected to a calendering treatment, and slit to a width of 1/2 inch to give a magnetic tape having a thickness of 17.5  $\mu\text{m}$ .

Table 1 shows the properties of the magnetic tapes obtained in the above manner in each Example.

Table 1

			Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5
Inert particles	A	Kind	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$
		Specific surface area	$\text{m}^2/\text{g}$	75	85	75	75
		Entire pore volume	$\text{ml/g}$	0.75	0.82	0.75	0.75
		Average particle diameter	$\mu\text{m}$	0.20	0.21	0.20	0.20
		Amount added	%	0.3	0.3	0.3	0.3
		Crystal form		$\theta$ form	$(\alpha+\gamma)$ form	$\theta$ form	$\theta$ form
	B	Kind	$\text{CaCO}_3$	$\text{CaCO}_3$	$\text{CaCO}_3$	$\text{SiO}_2$	kaolin
		Average particle diameter	$\mu\text{m}$	0.6	0.6	0.6	0.6
		Amount added	%	0.3	0.3	0.15	0.2
		Kind		-	-	$\text{SiO}_2$	-
		Average particle diameter	$\mu\text{m}$	-	-	0.6	-
		Amount added	%	-	-	0.15	-

- to be continued -



Table 1 (continued)

				Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5
Surface roughness Ra			nm	20	20	19	20	22
Running friction coefficient				0.20	0.20	0.17	0.16	0.25
Running friction coefficient increment				0.10	0.10	0.06	0.04	0.15
Projection distribution curve (Fig. 2)				A	-	B	C	D
Scratch resistance and abrasion resistance at high rate	Method A	Abrasion resistance		A	A	B	B	B
		Scratch resistance		A	A	A	A	A
	Method B	Abrasion resistance		A	A	B	B	B
		Scratch resistance		A	A	A	A	A
	Method C	Abrasion resistance		A	A	B	B	A
		Scratch resistance		A	A	A	A	A
Scratch resistance at low-rate repetition	Method A	Scratch resistance		A	A	A	A	B
	Method B	Scratch resistance		A	A	A	A	A
	Method C	Scratch resistance		A	A	A	B	A

As is clearly shown in Table 1, the biaxially oriented polyester film of the present invention not only has excellent lubricity as a substrate film for magnetic recording media but also has excellent abrasion resistance and scratch resistance against all of a metal tape guide having a well-finished surface, a metal tape guide having an insufficiently finished surface and a plastic tape guide. The biaxially oriented polyester film of the present invention has excellent properties as a substrate film for magnetic recording media.

#### 50 Claims

1. A biaxially oriented polyester film for magnetic recording media, which comprises:
  - (A) 0.05 to 1.0 % by weight of  $\theta$ -aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and
  - (B) 0.01 to 1.5 % by weight of inorganic particles other than  $\theta$ -aluminum oxide having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the  $\theta$ -aluminum oxide.

2. The biaxially oriented polyester film of Claim 1, wherein the  $\theta$ -aluminum oxide has an average particle diameter in the range of from 0.03 to 0.25  $\mu\text{m}$ .
3. The biaxially oriented polyester film of Claim 1, wherein the  $\theta$ -aluminum oxide has a specific surface area in the range of from 60 to 100  $\text{m}^2/\text{g}$ .
4. The biaxially oriented polyester film of Claim 1, wherein the  $\theta$ -aluminum oxide has an entire pore volume in the range of from 0.5 to 1.0  $\text{ml/g}$ .
5. The biaxially oriented polyester film of Claim 1, wherein the inert particles are inert inorganic particles or inert heat-resistant polymer particles.
6. The biaxially oriented polyester film of Claim 5, wherein the inert inorganic particles are formed of a material selected from silicon dioxide, titanium oxide, talc, kaolin, calcium carbonate and  $\alpha$ -,  $\delta$ -,  $\gamma$ -,  $\eta$ -,  $\rho$ -,  $\kappa$ - or  $\chi$ -aluminum oxide.
7. The biaxially oriented polyester film of Claim 5, wherein the inert heat-resistant polymer particles are selected from the group consisting of silicone resin particles, crosslinked acrylic particles, crosslinked polystyrene resin particles, fluorine resin particles and polyimide particles.
8. The biaxially oriented polyester film of Claim 1, wherein the inert particles have an average particle diameter in the range of from 0.2 to 1.2  $\mu\text{m}$ .
9. The biaxially oriented polyester film of Claim 1, wherein the polyester comprises an aromatic dicarboxylic acid as a main acid component and an aliphatic glycol having 2 to 10 carbon atoms as a main glycol component.
10. The biaxially oriented polyester film of Claim 1, which has a running friction coefficient ( $\mu\text{k}$ ) of not more than 0.3.
11. The biaxially oriented polyester film of Claim 1, which has a running friction coefficient increment ( $\Delta\mu\text{k}$ ) of not more than 0.15.
12. The biaxially oriented polyester film of Claim 1, which has projections satisfying the following relationship,  

$$-11.4x + 4 < \log y < -10.0x + 5$$
in which  $x$  is a distance ( $\mu\text{m}$ ) in the height direction from a standard level and  $y$  is a number of projections (pieces/ $\text{mm}^2$ ) counted when the projections are cut at a height of  $x$ , provided that  $x$  is in the range of not less than 0.05  $\mu\text{m}$  and that  $y$  is in the range of not less than 30/ $\text{mm}^2$ .
13. The biaxially oriented polyester film of Claim 1, which has a thickness in the range of from 4 to 100  $\mu\text{m}$ .
14. A magnetic recording medium comprising the biaxially oriented polyester film recited in Claim 1 and a magnetic layer formed thereon.
15. A biaxially oriented polyester film for magnetic recording media, which comprises:  
(A) 0.05 to 1.0 % by weight of mixed ( $\alpha+\gamma$ ) aluminum oxide having an average particle diameter in the range of from 0.02 to 0.3  $\mu\text{m}$  and a specific surface area in the range of from 50 to 120  $\text{m}^2/\text{g}$ , and  
(B) 0.01 to 1.5 % by weight of inert particles other than mixed ( $\alpha+\gamma$ ) aluminium oxide having an average particle diameter which is in the range of from 0.1 to 1.5  $\mu\text{m}$  and greater than the average particle diameter of the mixed ( $\alpha+\gamma$ ) aluminum oxide.
16. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha+\gamma$ ) aluminum oxide has an average particle diameter in the range of from 0.03 to 0.25  $\mu\text{m}$ .
17. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha+\gamma$ ) aluminum oxide has a specific surface area in the range of from 60 to 100  $\text{m}^2/\text{g}$ .
18. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha+\gamma$ ) aluminum oxide has an entire

pore volume in the range of from 0.5 to 1.0 ml/g.

19. The biaxially oriented polyester film of Claim 15, wherein the mixed ( $\alpha+\gamma$ ) aluminum oxide is formed of 3 to 30 % by weight of an  $\alpha$ -type crystal form and 70 to 97 % by weight of a  $\gamma$ -type crystal form.
20. A magnetic recording medium comprising the biaxially oriented polyester film recited in Claim 15 and a magnetic layer formed thereon.

## Patentansprüche

1. Biaxial orientierter Polyesterfilm für magnetische Aufzeichnungsträger, enthaltend:  
(A) 0,05 bis 1,0 Gew.-%  $\theta$ -Aluminiumoxid mit einem durchschnittlichen Teilchendurchmesser im Bereich von 0,02 bis 0,3  $\mu\text{m}$  und einer spezifischen Oberfläche im Bereich von 50 bis 120  $\text{m}^2/\text{g}$ , und  
(B) 0,01 bis 1,5 Gew.-% inerte Partikel anderen Typs als  $\theta$ -Aluminiumoxid mit einem durchschnittlichen Teilchendurchmesser, der im Bereich von 0,1 bis 1,5  $\mu\text{m}$  liegt und grösser ist als der durchschnittliche Teilchendurchmesser des  $\theta$ -Aluminiumoxids.
2. Biaxial orientierter Polyesterfilm nach Anspruch 1, bei dem das  $\theta$ -Aluminiumoxid einen durchschnittlichen Teilchendurchmesser im Bereich von 0,03 bis 0,25  $\mu\text{m}$  hat.
3. Biaxial orientierter Polyesterfilm nach Anspruch 1, bei dem das  $\theta$ -Aluminiumoxid eine spezifische Oberfläche im Bereich von 60 bis 120  $\text{m}^2/\text{g}$  hat.
4. Biaxial orientierter Polyesterfilm nach Anspruch 1, bei dem das  $\theta$ -Aluminiumoxid ein Gesamtporenvolumen im Bereich von 0,5 bis 1,0 ml/g hat.
5. Biaxial orientierter Polyesterfilm nach Anspruch 1, bei dem die inerten Partikel inerte anorganische Partikel oder inerte hitzebeständige Polymerpartikel sind.
6. Biaxial orientierter Polyesterfilm nach Anspruch 5, bei dem die inerten anorganischen Partikel aus einem Material bestehend aus Siliciumdioxid, Titanoxid, Talk, Kaolin, Calciumcarbonat oder  $\alpha$ -,  $\delta$ -,  $\gamma$ -,  $\eta$ -,  $\rho$ -,  $\kappa$ - oder  $\chi$ -Aluminiumoxid gebildet sind.
7. Biaxial orientierter Polyesterfilm nach Anspruch 5, bei dem die inerten hitzebeständigen Polymerpartikel ausgewählt sind aus der Gruppe bestehend aus Siliciumharzpartikeln, vernetzten Acrylpartikeln, vernetzten Polystyrolharzpartikeln, Fluorharzpartikeln und Polyimidpartikeln.
8. Biaxial orientierter Polyesterfilm nach Anspruch 1, bei dem die inerten Partikel einen durchschnittlichen Teilchendurchmesser im Bereich von 0,2 bis 1,2  $\mu\text{m}$  haben.
9. Biaxial orientierter Polyesterfilm nach Anspruch 1, bei dem der Polyester eine aromatische Dicarbonsäure als eine Hauptsäurekomponente und ein aliphatisches Glykol mit 2 bis 10 Kohlenstoffatomen als eine Hauptglykolkomponente aufweist.
10. Biaxial orientierter Polyesterfilm nach Anspruch 1, der einen Lauffreibungskoeffizient ( $\mu\text{k}$ ) von nicht mehr als 0,3 hat.
11. Biaxial orientierter Polyesterfilm nach Anspruch 1, der ein Lauffreibungs-koeffizientinkrement ( $\Delta\mu\text{k}$ ) von nicht mehr als 0,15 hat.
12. Biaxial orientierter Polyesterfilm nach Anspruch 1, der Vorsprünge hat, die der folgenden Beziehung genügen

$$-11,4x + 4 < \log y < -10,0x + 5$$

worin x einen Abstand ( $\mu\text{m}$ ) in der Höhenrichtung von einem Standardlevel und y eine Zahl von Vorsprüngen (Anzahl/ $\text{mm}^2$ ), berechnet, wenn die Vorsprünge bei einer Höhe von x abgeschnitten sind, bedeutet n, mit der Maßgabe, daß x im Bereich von nicht weniger als 0,05  $\mu\text{m}$  und daß y im Bereich von nicht weniger

als 30/mm<sup>2</sup> liegen.

13. Biaxial orientierter Polyesterfilm nach Anspruch 1, der eine Dicke im Bereich von 4 bis 100 µm hat.
- 5 14. Magnetischer Aufzeichnungsträger, der den biaxial orientierten Polyesterfilm gemäß Anspruch 1 und eine darauf aufgebrachte magnetische Schicht aufweist.
- 10 15. Biaxial orientierter Polyesterfilm für magnetische Aufzeichnungsträger, welcher aufweist:  
 (A)' 0,05 bis 1,0 Gew.-% ( $\alpha + \gamma$ )-Aluminiumoxidgemisch mit einem durchschnittlichen Teilchendurchmesser im Bereich von 0,02 bis 0,3 µm und einer spezifischen Oberfläche im Bereich von 50 bis 120 m<sup>2</sup>/g, und  
 (B)' 0,01 bis 1,5 Gew.-% inerte Partikel anderen Typs als ( $\alpha + \gamma$ )-Aluminiumoxidgemisch mit einem durchschnittlichen Teilchendurchmesser, der im Bereich von 0,1 bis 1,5 µm liegt und größer als der durchschnittliche Teilchendurchmesser des ( $\alpha + \gamma$ )-Aluminiumoxidgemisches ist.
- 15 16. Biaxial orientierter Polyesterfilm nach Anspruch 15, bei dem das ( $\alpha + \gamma$ )-Aluminiumoxidgemisch einen durchschnittlichen Teilchendurchmesser im Bereich von 0,03 bis 0,25 µm hat.
- 20 17. Biaxial orientierter Polyesterfilm nach Anspruch 15, bei dem das ( $\alpha + \gamma$ )-Aluminiumoxidgemisch eine spezifische Oberfläche im Bereich von 60 bis 100 m<sup>2</sup>/g hat.
18. Biaxial orientierter Polyesterfilm nach Anspruch 15, bei dem das ( $\alpha + \gamma$ )-Aluminiumoxidgemisch ein Gesamtporenvolumen im Bereich von 0,5 bis 1,0 ml/g hat.
- 25 19. Biaxial orientierter Polyesterfilm nach Anspruch 15, bei dem das ( $\alpha + \gamma$ )-Aluminiumoxidgemisch aus 3 bis 30 Gew.-% einer  $\alpha$ -Typ-Kristallform und 70 bis 97 Gew.-% einer  $\gamma$ -Typ-Kristallform gebildet ist.
- 30 20. Magnetischer Aufzeichnungsträger, der den biaxial orientierten Polyesterfilm gemäß Anspruch 15 und eine darauf aufgebrachte magnetische Schicht aufweist.

#### Revendications

- 35 1. Film polyester à orientation bi-axiale pour milieux d'enregistrement magnétique, comprenant:  
 (A) 0,05 à 1,0% en poids d'oxyde d'aluminium  $\theta$  ayant un diamètre moyen de particule compris entre 0,02 et 0,3 µm et une aire de surface spécifique comprise entre 50 et 120 m<sup>2</sup>/g, et  
 (B) 0,01 à 1,5% en poids de particules inertes autres que l'oxyde d'aluminium  $\theta$  ayant un diamètre moyen de particule situé entre 0,1 et 1,5 µm, et supérieur au diamètre moyen de particule de l'oxyde d'aluminium  $\theta$ .
- 40 2. Film polyester à orientation bi-axiale selon la revendication 1, dans lequel l'oxyde d'aluminium  $\theta$  possède un diamètre moyen de particule compris entre 0,03 et 0,25 µm.
- 45 3. Film polyester à orientation bi-axiale selon la revendication 1, dans lequel l'oxyde d'aluminium  $\theta$  possède une aire de surface spécifique comprise entre 60 et 100 m<sup>2</sup>/g.
4. Film polyester à orientation bi-axiale selon la revendication 1, dans lequel l'oxyde d'aluminium  $\theta$  possède un volume total de pores compris entre 0,5 et 1,0 ml/g.
- 50 5. Film polyester à orientation bi-axiale selon la revendication 1, dans lequel les particules inertes sont des particules inorganiques inertes ou des particules de polymère inerte résistant à la chaleur.
- 55 6. Film polyester à orientation bi-axiale selon la revendication 5, dans lequel les particules inorganiques inertes sont formées d'une substance choisie parmi le dioxyde de silicium, l'oxyde de titane, le talc, l kaolin, le carbonate de calcium et l'oxyde d'aluminium  $\alpha$ ,  $\delta$ ,  $\gamma$ ,  $\eta$ ,  $\rho$ ,  $\kappa$  ou  $\chi$ .
7. Film polyester à orientation bi-axiale selon la revendication 5, dans lequel les particules de polymère inerte résistant à la chaleur, sont choisies parmi le groupe constitué par des particules de résin de silicium ,

des particules acryliques réticulées, des particules de résine de polystyrène réticulé, des particules de résines fluorées et des particules de polyimide.

- 5 8. Film polyester à orientation bi-axiale selon la revendication 1, dans lequel les particules inertes ont un diamètre moyen de particule compris entre 0,2 et 1,2  $\mu\text{m}$ .
9. Film polyester à orientation bi-axiale selon la revendication 1, dans lequel le polyester comprend un acide dicarboxylique aromatique comme composant acide principal et un glycol aliphatique ayant de 2 à 10 atomes de carbone comme composant glycol principal.
- 10 10. Film polyester à orientation bi-axiale selon la revendication 1, possédant un coefficient de frottement de roulement ( $\mu\text{k}$ ) non supérieur à 0,3.
11. Film polyester à orientation bi-axiale selon la revendication 1, possédant un accroissement de coefficient de frottement de roulement ( $\Delta\mu\text{k}$ ) non supérieur à 0,15.
12. Film polyester à orientation bi-axiale selon la revendication 1, ayant des protubérances satisfaisant à la relation suivante,
 
$$-11,4x + 4 < \log y < -10,0x + 5$$
 20 dans laquelle x est une distance ( $\mu\text{m}$ ) selon la direction verticale à partir d'un niveau de référence et y est un nombre de protubérances (éléments/ $\text{mm}^2$ ) compté lorsque les protubérances sont coupées à une hauteur de x, à condition que x ne soit pas inférieur à 0,05  $\mu\text{m}$  et que y ne soit pas inférieur à 30/ $\text{mm}^2$ .
- 25 13. Film polyester à orientation bi-axiale selon la revendication 1, possédant une épaisseur comprise entre 4 et 100  $\mu\text{m}$ .
14. Milieu d'enregistrement magnétique comprenant le film polyester à orientation bi-axiale énoncé dans la revendication 1 et une couche magnétique formée sur celui-ci.
- 30 15. Film polyester à orientation bi-axiale pour milieux d'enregistrement magnétique, qui comprend:
 (A) 0,05 à 1,0% en poids d'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ) ayant un diamètre moyen de particule compris entre 0,02 et 0,3  $\mu\text{m}$  et une aire de surface spécifique comprise entre 50 et 120  $\text{m}^2/\text{g}$ , et
 (B) 0,01 à 1,5% en poids de particules inertes autres que l'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ) ayant un diamètre moyen de particule compris entre 0,1 et 1,5  $\mu\text{m}$ , et supérieur au diamètre moyen de particule de l'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ).
- 35 16. Film polyester à orientation bi-axiale selon la revendication 15, dans lequel l'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ) possède un diamètre moyen de particule compris entre 0,03 et 0,25  $\mu\text{m}$ .
- 40 17. Film polyester à orientation bi-axiale selon la revendication 15, dans lequel l'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ) possède une aire de surface spécifique comprise entre 60 et 100  $\text{m}^2/\text{g}$ .
- 45 18. Film polyester à orientation bi-axiale selon la revendication 15, dans lequel l'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ) possède un volume global de pores compris entre 0,5 et 1,0  $\text{ml/g}$ .
19. Film polyester à orientation bi-axiale selon la revendication 15, dans lequel l'oxyde d'aluminium mélangé ( $\alpha+\gamma$ ) est formé de 3 à 30% en poids d'une forme cristalline de type  $\alpha$  et de 70 à 97% en poids d'une forme cristalline de type  $\gamma$ .
- 50 20. Milieu d'enregistrement magnétique comprenant le film polyester à orientation bi-axiale énoncé dans la revendication 15 et une couche magnétique formée sur celui-ci.

FIG. 1

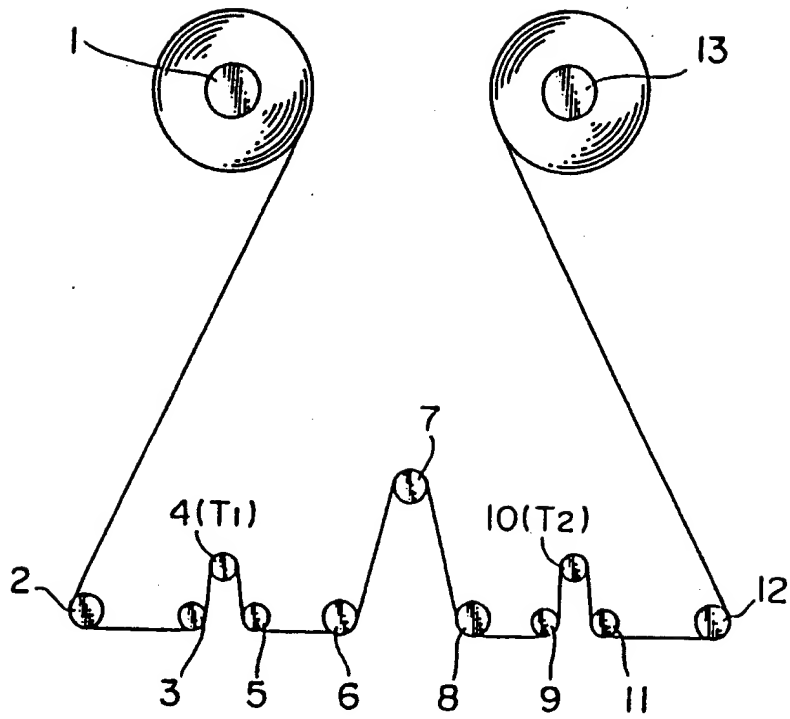


FIG. 2

